HIGH STRENGTH, HIGH TOUGHNESS IN SITU CERAMIC COMPOSITES

Progress Report for the Quarter:
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Task 1: Composite Synthesis

Composite synthesis efforts during this period were directed: (a) to improve the composite's bulk density, and (b) to understand the displacement reaction process. A higher hot pressing pressure (45 MPa) was used to increase the composite density. The oxygen content of the TiC powder and hot pressing schedule were varied to observe their effects on the displacement reaction and types of phases formed.

It was reported in the previous progress report that Ti_3SiC_2-SiC composites can be formed by pressureless sintering at 1300°C. However, TiC, Si, and $TiSi_2$ phases were also detected in these samples in addition to Ti_3SiC_2 and SiC phases. It was thought that the reaction did not go to completion due to the lack of physical contact between the reactant powder particles. X-ray diffraction spectra of the sample hot pressed at 1300°C (under 30 MPa pressure for 2 hours) showed only Ti_3SiC_2-SiC phase mixture. Thus, it is critical that physical contact must be maintained for reaction to be completed. Although hot pressing at 1300°C gave a pure Ti_3SiC_2-SiC phase mixture, the sample was only densified to 89% of theoretical density. Therefore, either higher temperatures than 1300°C, or higher pressures *and* temperatures are required to improve bulk density. Optimization of the hot pressing temperature and pressure should be investigated in the Phase II study to improve the composite properties.

It was found that the oxygen content in TiC powders plays an important role in composite synthesis. While the presence of oxygen improves the composite density, it also tends to decrease the reaction rate. This finding is supported by the following results.

Chemical analysis of the three different TiC and Si powder batches are shown in Table 1. The Batch A powders were used by QUEST during the previous progress report period. The Batch B mixture was prepared by Battelle and mechanical test results from this batch were reported in the previous progress report. Batch C is the recently purchased powders with low oxygen content. Table 2 shows the hot pressing conditions, resulting density, and phase compositions for the samples prepared from these three powder batches.

Composite density increased with increasing pressing pressure, as expected. The presence of oxygen in the powder mixture appears to help densification. However, the samples contained some unreacted TiC in addition to $TiSi_2$ which may be an intermediate phase towards Ti_3SiC_2 formation. This observation suggests that oxygen decreases the reaction rate and slows down SiC formation. Thus there is sufficient time for the material to densify before SiC phase grow inside the Ti_3SiC_2 matrix. In low oxygen containing samples, SiC phase may be forming at a much faster rate so the composite can not be densified as easily, since the presence of SiC particles hinders densification. The reaction rate should be controlled to control the microstructural development and densification. This aspect of the research will be addressed in more detail in the remainder of the Phase I program and further in the Phase II program.

Task 2: Composite Characterization

Wear tests were performed on 32 compositions (Samples 32-B, 32-31, and 32-45 in Table 2) and 33 compositions (Sample 33 in Table 2) at Argon National Laboratories. The ball-on-disk test configuration with a 440 steel ball (hardness $R_C = 63$) was used. The tests were conducted on 20 × 20 × 5 mm^3 size samples in air at room temperature. Sample surfaces were polished with diamond 0.25 μm paste. A load of 2N was applied on the ball, and the ball was slided across the specimen at a speed of 0.05m/sec. A total of 100 meters sliding distance was used in each test.

The wear of steel ball for each composite was measured and is given in Figure 1. As Figure 1 shows, sample 32-B with low oxygen content causes the highest ball wear. This behavior is attributed to the higher SiC content and higher porosity of this sample, since the amount of hard phase and porosity can affect the wear properties.

As the pressing pressure increased from 31 MPa to 45 MPa for the 32 Composition, ball wear decreased. This behavior is explained by elimination of porosity due to improved densification. Ball wear decreased with increasing density for 32 samples, as shown in Figure 2. Ball wear for the 33 composition (Sample 33, d = 4.07 g/cc) was also found to be very low. However, processing difficulties need to be addressed for this composition, as reported in the previous progress report.

The friction coefficient of the samples against the steel ball were found to be between 0.6 and 0.8, as shown in Figure 3. Friction coefficients started out low (0.2), but increased and became constant with time. This is normal since the composite is harder than steel, a steel layer forms on the composite as the test progresses, and steel starts rubbing steel, effectively.

Flexural strength test specimens from samples 32-31, 32-45, 32-C, 32-CT (see Table 2) were already prepared. Room and high temperature mechanical tests are currently being conducted to determine composite strength.

Schedule

The program is on schedule without any delays. As of April 29, 1996, 80% of the project budget has been spent, and 10% is obligated.

Future Work

The following activities are planned for the next reporting period:

1. Finish mechanical testing of samples at room and high temperature.
2. Characterize the microstructure using light and electron microscopy.
3. Prepare the final report with recommendations for the Phase II study.

Table 1: Carbon, Nitrogen, and Oxygen Analysis of Powders Used for Ti_3SiC_2-SiC Composites.

Batch	TiC (%)			Si (%)	
	C	N	O	N	O
A	19.10	0.07	1.38	0.004	0.18
B	19.57	0.02	0.13	0.003	0.50
C	19.50	0.12	0.13	0.002	0.19

Table 2: Composite Density and Phases Detected in Ti_3SiC_2-SiC Samples Prepared from Different TiC-Si Powder Mixtures.

		Hot Pressing Conditions				
Sample	Powder Batch	Temp. (°C)	Pressure (MPa)	Time (min.)	Density (g/cc)	Phases
32-B	B	1500	30	30	4.09	Ti_3SiC_2, SiC
32-31	A	1500	30	30	4.11	Ti_3SiC_2, SiC, TiC, $TiSi_2$
32-45	A	1500	45	45	4.23	Ti_3SiC_2, SiC, TiC, $TiSi_2$
32-C	C	1500	45	45	4.11	not yet determined
32-CT	C+TiO_2	1500	45	45	4.27	not yet determined
33	A	1500	30	30	4.07	Ti_3SiC_2, SiC, TiC, $TiSi_2$

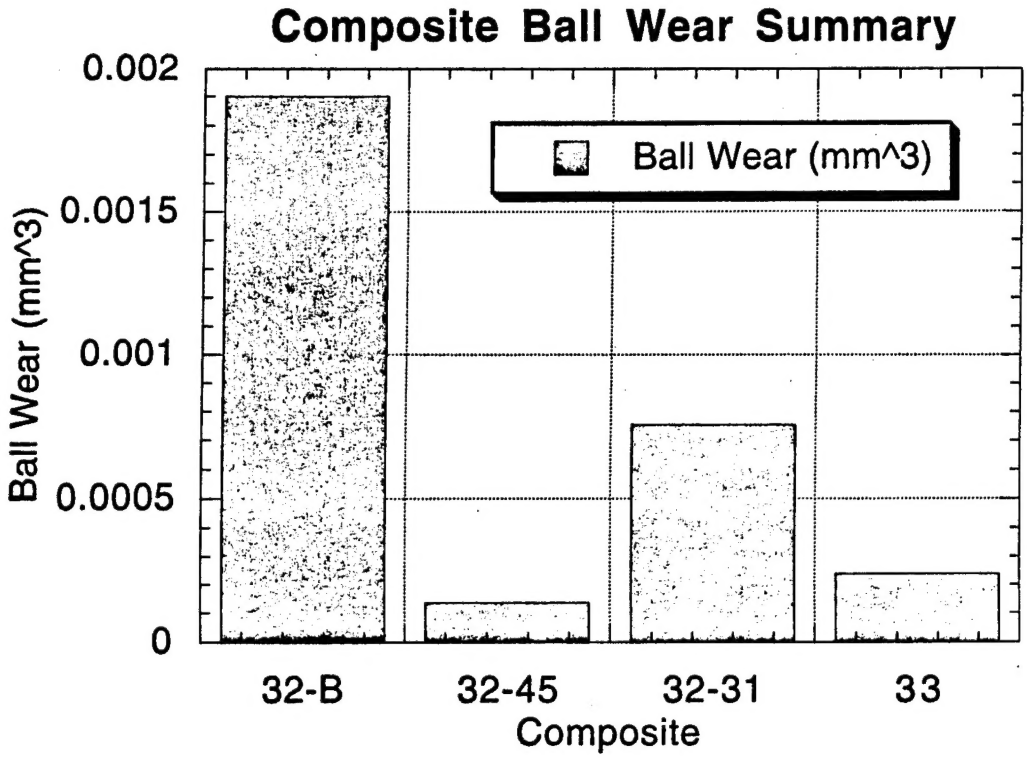

Figure 1 - Composite Ball Wear Test Summary.

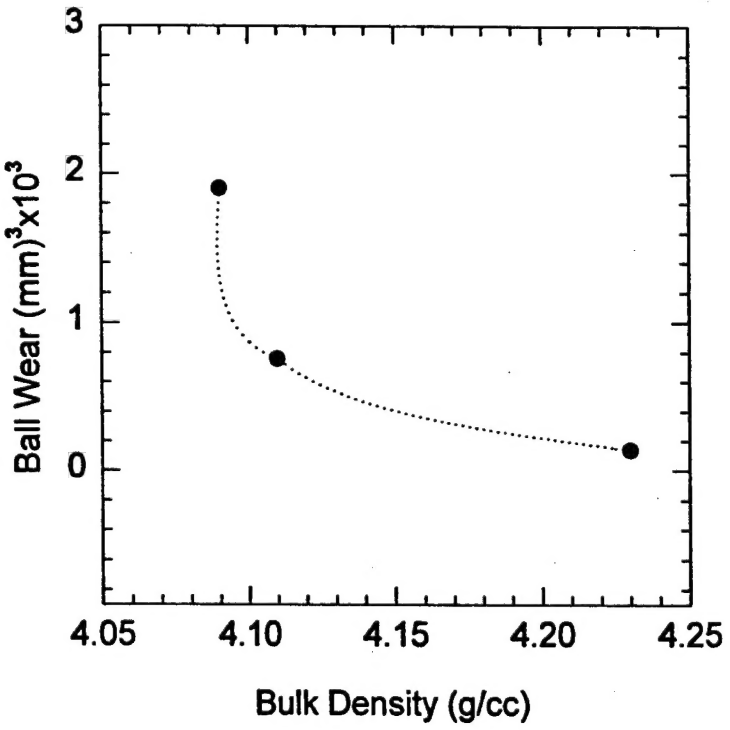

Figure 2 - The Effect of Composite Density on the Ball Wear.

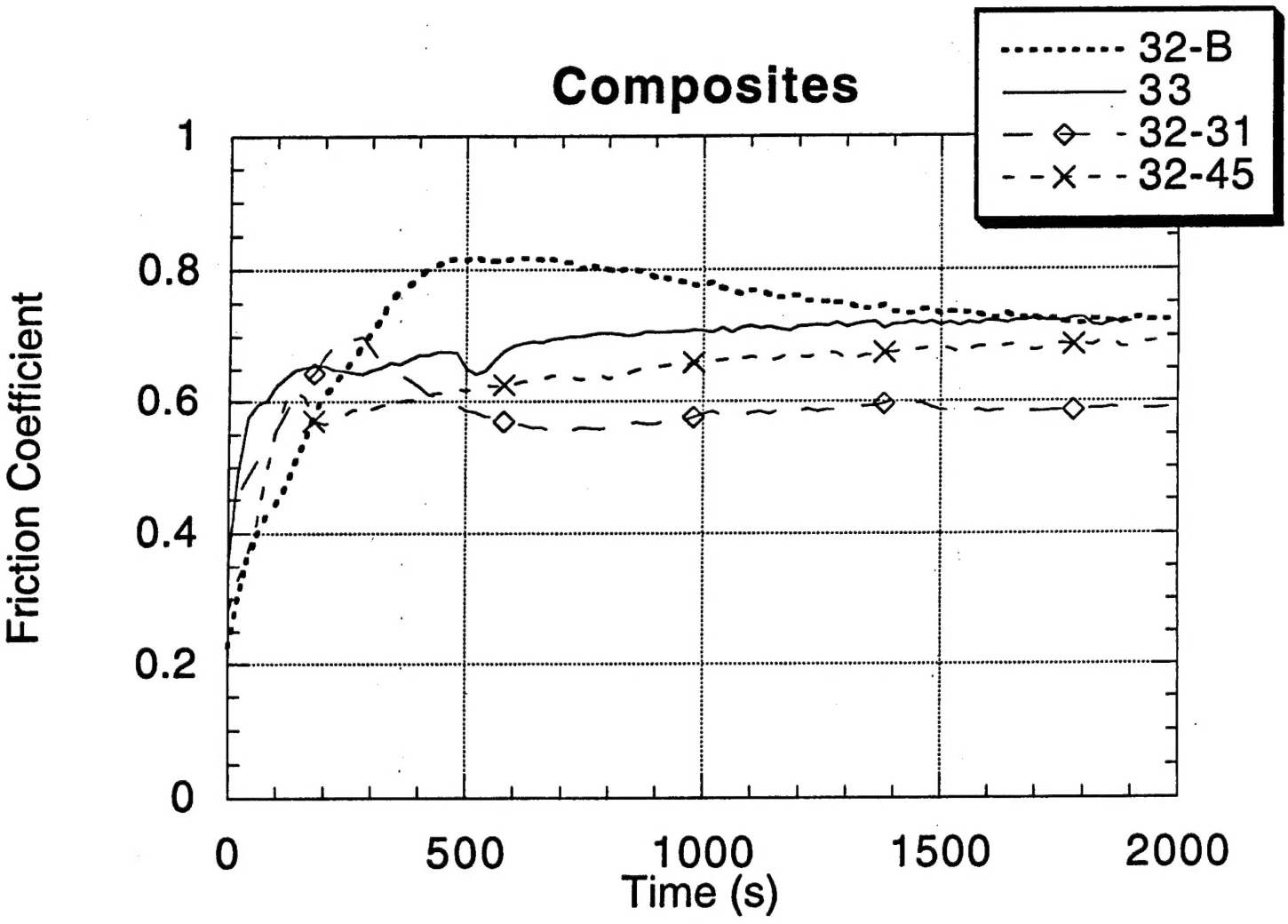

Figure 3 - Friction Coefficients Between the Composites and 440 Steel.